

and it is an object of the present invention to provide an aluminum nitride sintered body having a large area and a small thickness and also having the property of a controlled warp and waviness and further to provide a metallized substrate and a heater using the aluminum nitride sintered body. Another
5 object of the present invention is to provide methods of producing such an aluminum nitride sintered body.

Further, another object of the present invention is to provide a jig used in the above method of producing the aluminum nitride sintered body.

The aluminum nitride sintered body according to a first aspect of the
10 present invention has a maximum length of 320 mm or more, a thickness of more than 0 mm and 2 mm or less, a warpage of 0 $\mu\text{m}/\text{mm}$ or more and less than 2 $\mu\text{m}/\text{mm}$, and a local waviness height of 0 μm or more and 100 μm or less.

In this manner, the aluminum nitride sintered body having a large
15 area and having small warpage and waviness is suitable to be used as the substrate for electronic parts, etc. That is, when the substrate made of the aluminum nitride sintered body with large dimensions is used as a material for constructing electronic parts such as a metallized substrate and a heater, the metallized substrate and the heater having a large area can be realized.
20 Further, when manufacturing the metallized substrate and heater with a small size, a large number of metallized substrates and heaters are obtainable from a single substrate by dividing a substrate made of the aluminum nitride sintered body according to the present invention into parts.

the aluminum nitride sintered body is preferably 1.5 mm or less and more preferably 1.0 mm or less.

In addition, if the warpage is 0 $\mu\text{m}/\text{mm}$ or more and less than 2 $\mu\text{m}/\text{mm}$ and the local waviness height is 0 μm or more and 100 μm or less, the
5 sufficient flatness of the aluminum nitride substrate can be ensured. Therefore, it is possible to prevent the occurrence of defects in the screen printing method as described above. Also, the warpage is preferably less than 1.5 $\mu\text{m}/\text{mm}$ and more preferably less than 1.0 $\mu\text{m}/\text{mm}$. The waviness height is preferably 75 μm or less, and more preferably 50 μm or less.

10 The aluminum nitride sintered body according to the first aspect of the present invention may have the thermal conductivity ranging from 50 W/m · K to 250 W/m · K.

In such a case, when the present aluminum nitride sintered body is used as a substrate of a heater, it is possible to achieve proper heat diffusion
15 over the entire portion of the substrate and to reduce the risk of other members (such as electrode part or control circuit, etc.) formed on the substrate being affected adversely by heat generated from a heating element (the heating element formed on the surface of the substrate) constituting the heater. If the thermal conductivity of the aluminum nitride sintered body is
20 less than 50 W/m · K, thermal conductivity of the substrate made of the aluminum nitride sintered body is excessively small and hence heat diffusion from the heating element over the entire substrate becomes insufficient, thereby making a difficulty to obtain a uniform distribution of heat over the

entire portion of the substrate. Also, if the thermal conductivity of the aluminum nitride sintered body exceeds $250 \text{ W/m} \cdot \text{K}$, it may cause a risk of overheating of other members such as the electrode part and control circuits, etc. mounted on the substrate, due to the heat from the heating element. As
5 a result thereof, the other members are at risk of damage due to overheating.

The thermal conductivity of the aluminum nitride sintered body is preferably in the range of $80 \text{ W/m} \cdot \text{K}$ to $200 \text{ W/m} \cdot \text{K}$ and, and more preferably in the range of $85 \text{ W/m} \cdot \text{K}$ to $105 \text{ W/m} \cdot \text{K}$.

A metallized substrate according to another (the second) aspect of the
10 present invention comprises a substrate and a metallized layer. The substrate is a substrate made of the aluminum nitride sintered body formed according to the first aspect of the present invention and has a plate shape (a sheet shape). The metallized layer is typically formed on at least a part of the surface of the substrate. And the metallized layer has electric conductivity.

15 The metallized substrate according to another (the third) aspect of the present invention comprises a substrate including the aluminum nitride sintered body and a metallized layer. The substrate has a maximum length of 320 mm or more, a thickness of more than 0 mm and 2 mm or less, and the local waviness height of $0 \text{ } \mu\text{m}$ or more and $100 \text{ } \mu\text{m}$ or less. The metallized
20 layer is formed on at least a part of the surface of the substrate. The metallized layer has electric conductivity. The metallized substrate according to the above aspect of the present invention has the warpage value ranging from $0 \text{ } \mu\text{m/mm}$ to $5 \text{ } \mu\text{m/mm}$. Also, the metallized layer may be a layer

containing metal or a layer comprising other materials with electric conductivity.

Thus, it is possible to obtain metallized substrates with larger dimensions and superior flatness as compared with conventional metallized
5 substrates. Therefore, by using the metallized substrate of the present invention, it is possible to produce electronic parts with large sizes. Further, in the case of producing electronic parts with relatively small sizes by dividing the metallized substrate according to the present invention, it is possible to increase the number of the electronic parts, thereby reducing costs for
10 producing the electronic parts.

Further, if the substrate forming the metallized substrate has the maximum length of 320 mm or more, it is possible to obtain metallized substrates with larger dimensions than conventional ones. Also, if the substrate has the thickness of more than 0 mm and 2 mm or less, it is suitable
15 for use as a metallized substrate and hence shows excellent properties as the substrate of an electronic apparatus, the substrate for a heater, etc. Further, in the case where the local waviness height is within a range of 0 μm or more and 100 μm or less and the warp is within a range of 0 $\mu\text{m}/\text{mm}$ or more and 5 $\mu\text{m}/\text{mm}$ or less, it is possible to reduce the probable occurrence of accidents
20 such as cracking of the substrate even in the case where the metallized substrate or the heater is employed under a condition of applied mechanical load (load).

In addition, the aluminum nitride sintered body according to the first

aspect of the present invention and the aluminum nitride sintered body to construct the substrate contained in the metallized substrate according to the other (the third) aspect of the present invention are preferably produced according to the following procedure. That is, before a step to remove a
5 binder included in a molded body that is to become the aluminum nitride sintered body (the binder removing step), a step of naturally drying the molded body for 1 hour or more is performed, followed by the above binder removing step and a sintering step. The time required for the natural drying is more preferably 10 hours or more, and most preferably 20 hours or more. The
10 aluminum nitride sintered body is preferably produced by arranging the molded body to form the aluminum nitride sintered body in a space surrounded by a jig principally comprising boron nitride; then sintering the arranged body under a condition where the ratio of the volume of the molded body before sintering to the volume of the space is neither less than 10% nor
15 more than 70%.

The heater according to another aspect (the fourth) of the present invention comprises the metallized substrate according to the second aspect or the third aspect of the present invention, an electrode part, and an insulating layer. The electrode part is arranged on the surface of the substrate which
20 constitutes the metallized substrate and is connected to the metallized layer. The insulating layer is provided on the top surface of the metallized layer. The metallized layer functions as a heating element by being supplied with electrical current from the electrode part.

Thus, by using the metallized substrate of the present invention, it is possible to produce a heater having a larger area and superior flatness as compared with conventional heaters. Also, by dividing the heater of the present invention into parts, heaters of a relatively smaller size can be produced. In such a case, since the number of the heaters obtainable from a single metallized substrate can be increased, the production cost of the heaters can be reduced.

A method of producing the aluminum nitride sintered body according to another (the fifth) aspect of the present invention comprises a step of preparing a raw material containing a binder and aluminum nitride as the major raw material. The method further comprises a step of producing a sheet-shaped molded body by using the raw material. Further, a drying step of naturally drying the molded body for 1 hour or more is performed, followed by a step of removing the binder from the dried molded body. Afterward, a sintering step is performed so that the molded body free of the binder is sintered. In the drying step, the molded body is preferably dried under natural conditions for 1 hour or more. The natural drying is performed more preferably for 10 hours or more, and most preferably for 20 hours or more.

By performing the above procedures, because the drying step is conducted between the step of producing the molded body and the step of removing the binder (the binder removing step), a solvent, moisture, etc. slowly and sufficiently volatilize out of the whole molded body by the drying step. Thus, the molded body contracts almost uniformly over the entire

portion thereof as a result of volatilization of the solvent and moisture. As a result, the generation of distortion and internal stress caused by the volatilization of the solvent and moisture can be controlled. Accordingly, during the binder removing step and the sintering step as post-steps, it is possible to prevent deformation such as warpage or waviness in the molded body or the sintered body.

In addition, when the drying time in the drying step (the natural drying time) is 1 hour or more, moisture or the like can be uniformly and sufficiently volatilized out of the entire portion of the molded body at relatively low speed.

With respect to the method of producing the aluminum nitride sintered body according to the fifth aspect of the present invention, the sintering step comprises sintering the molded body under a condition that the molded body is arranged in the space surrounded by the jig comprising boron nitride as the major component. Preferably, the ratio of the volume of the molded body before sintering to the volume of the space surrounded by the above jig ranges from 10% to 70%.

In this case, since the molded body is sintered in a state of being arranged within the space surrounded (substantially closed) by the jig, it is possible to reduce a risk of generating a local flow of atmospheric gas near the molded body during the sintering step. Thus, the risk of adverse effects on the shape of the molded body (the sintered body) caused depending on the condition of the atmospheric gas can be reduced.

space is within the range described above, the partial pressures of components of the atmospheric gas around the molded body can properly be adjusted during the sintering step.

5 Brief Description of Drawings

Figure 1 is a schematic perspective view illustrating embodiment 1 of an aluminum nitride substrate according to the present invention;

Figure 2 is a schematic sectional view explaining the waviness height of the aluminum nitride substrate shown in Fig. 1;

10 Figure 3 is a schematic perspective view explaining the warpage of the aluminum nitride substrate shown in Fig. 1;

Figure 4 shows a flow chart explaining the method of producing the aluminum nitride substrate shown in Fig. 1;

15 Figure 5 is a schematic perspective view illustrating a jig used in the sintering step shown in Fig. 4;

Figure 6 is a schematic plane view illustrating a state in which a molded body is mounted in the depressed portion of the jig before the sintering step;

20 Figure 7 is a schematic perspective view illustrating a jig in which a molded body mounted as shown in Fig. 6 is being stacked onto another of such a jig;

Figure 8 is a schematic sectional view illustrating the jig pile formed by piling up a plurality of jigs as shown in Fig. 7;

Figure 9 is an enlarged schematic view illustrating one of the jigs of the jig pile shown in Fig. 8;

Figure 10 is a schematic perspective view illustrating a state of the jig pile shown in Fig. 8 that is placed in a metal case;

5 Figure 11 is a schematic sectional view illustrating a metallized substrate according to the present invention;

Figure 12 is a schematic plane view illustrating a heater according to the present invention;

10 Figure 13 is a schematic sectional view taken along the line XIII-XIII of Fig. 12; and

Figure 14 is a schematic sectional view explaining a screen printing method used to form the heating element and the electrode part on the surface of each sample (substrate).

15 Best Mode for Carrying Out the Invention

Hereafter, the present invention will be described in more detail by reference to the accompanying figures. In the figures, the same reference numeral is attached to the same or equivalent part, and the explanation thereof is not repeated.

20 (First embodiment)

Figure 1 is a schematic perspective view illustrating a first embodiment of an aluminum nitride substrate according to the present invention. Figure 2 is a schematic sectional view explaining the waviness

height of the aluminum nitride substrate shown in Fig. 1. Figure 3 is a schematic perspective view illustrating the warpage of the aluminum nitride substrate shown in Fig. 1. With reference to Figs. 1 to 3, the first embodiment of the present invention for the aluminum nitride substrate will
5 be described in detail.

As shown in Fig. 1, the aluminum nitride substrate 1 as an aluminum nitride sintered body according to the present invention comprises a sintered body comprising aluminum nitride as a major component thereof and having a rectangular form with length L, width W and thickness T. Maximum length
10 ML of the aluminum nitride substrate 1 (hereinafter also referred to as "substrate 1") corresponds to a length of a diagonal line on the top surface of the substrate 1.

The maximum length ML of the substrate 1 is equal to 320 mm or more. In this manner, a substrate for electronic apparatuses or a metallized
15 substrate which is greater than conventional ones can be realized. The maximum length ML is preferably not less than 350 mm and, more preferably, 450 mm or more. The thickness T of the substrate 1 is more than 0 mm and 2 mm or less. With this arrangement, the substrate 1 of the present invention can easily be applied to the substrate for a heater or the like. Further, the
20 thickness T is preferably 1.5 mm or less and, more preferably, 1.0 mm or less.

In addition, the waviness height (hereinafter also referred to as waviness) of the substrate 1 shown in Fig. 1 ranges from 0 μ m or more to 100 μ m or less. For the substrate 1, the waviness height is preferably 75 μ m or

less, and more preferably 50 μm or less. As shown in Fig. 2, the waviness height used here refers to a height H of a locally convex portion 2 (a portion waved protruding at the outer surface) on the surface of the substrate 1, wherein the height H is the highest height measured from a substrate surface 3 outside the locally convex portions 2 up to the highest part of the convex portion 2 that is the largest convex (the highest height H) among the displacements measured at a plurality of points of the surface.

The waviness height can easily be determined by measuring the displacement of the surface of the substrate 1 by using a laser displacement meter or a probe type displacement meter. In addition, in the substrate 1, the warpage ranges from 0 $\mu\text{m}/\text{mm}$ or more to less than 2 $\mu\text{m}/\text{mm}$. The warpage is preferably less than 1.5 $\mu\text{m}/\text{mm}$ and, more preferably, less than 1.0 $\mu\text{m}/\text{mm}$. Here, the warpage can be obtained as described below. That is, as shown in Fig. 3, the displacement of the surface of the substrate 1 is measured along the routes of arrows 4a to 4d by using the laser displacement meter or the probe type displacement meter. Then, from the results of such measurements, the difference between the lowest portion and the highest portion of the surface of the substrate 1 is obtained. Further, the difference is divided by the maximum length ML of the substrate 1 (Fig. 1). As a result, the value of warpage of the substrate 1 is obtained.

As described above, the substrate 1 according to the present invention has a small amount of warpage and waviness height (waviness) despite its large size. In other words, the substrate 1 of the present invention has a

large area and simultaneously excellent flatness. Here, a case is considered wherein the substrate 1 is applied to a substrate to construct electronic parts such as a laser printer and/or a heater in a copying machine. In the case where an electrode or heating element of the heater, etc. is to be formed on the surface of the substrate 1 by using a screen printing method, the heating element or the like cannot occasionally be formed properly on the substrate 1 if a warp or the like of the substrate 1 exists. However, with the substrate 1 of the present invention, it is possible to accurately print an electric conductive paste (which is to be formed as a heating element) or the like by using the screen printing method, because the substrate 1 has excellent flatness. Therefore, it is possible to prevent the occurrence of problems such as formation failure of the heating element or the like.

Further, if the substrate 1 is warped or curved during screen printing, problems may occur such as cracks in the substrate 1 or damage of the screen. However, by using the substrate 1 of the present invention, it is possible to prevent the occurrence of such problems. As a result, it is possible to reduce the cost of production of the heater or the like.

Further, if the substrate 1 of the present invention is employed, a metallized substrate or a heater having a large area can easily be produced. The plane form of the substrate 1 may not be limited to the rectangular form as shown in Fig. 1.

The substrate 1 according to an embodiment of the present invention shown in Fig. 1 has a thermal conductivity within a range of 50 W/m · K to 250

W/m·K. Also, the aluminum nitride sintered body of the present invention can adopt a bar-shaped form. The thermal conductivity is preferably in the range of 80 W/m·K to 200 W/m·K and, more preferably in the range of 85 W/m·K to 105 W/m·K. In this range, if the substrate 1 shown in Fig. 1 is applied to the heater, heat diffusion over the entire portion of the substrate 1 can be made sufficiently fast, and at the same time it is possible to prevent the occurrence of problems such that the electrode part which supplies electric current to the heating element constituting the heater is damaged by heat from the heater.

Herein, if the thermal conductivity is less than 50 W/m·K, heat diffusion speed of the substrate 1 becomes insufficient and it is difficult to maintain the temperature of the whole of the substrate 1 uniformly. Also, in the case where the thermal conductivity exceeds 250 W/m·K, the heat diffusion speed is so fast that, when the substrate 1 is used as the substrate of the heater, the heat generated in the heating element is transferred easily to the substrate 1, and further to the electrode part which supplies electric current to the heating element and to other peripheral circuit parts formed on the surface of the substrate. Thus, due to overheating of the electrode part by the heat generated in the heating element, there is the possibility that damage is caused by the heat to lead wires connected to the electrode part and to the peripheral circuit parts, etc. arranged around the electrode part.

The surface roughness of the substrate 1 is defined as Ra and, preferably 1.0 μm or less, and more preferably 0.4 μm or less. With such Ra,

when the heating element to constitute the heater or the metallized layer or the like is formed on the surface of the substrate 1, the adhesiveness between the heating element or the metallized layer and the surface of the substrate 1 can be enhanced.

5 Also, the transverse rupture strength of the substrate 1 is preferably 200 MPa or more in terms of the three-point bending strength. With this strength, in the case where the surface of the substrate 1 is treated for forming the metallized layer or the like, and in the case where the metallized substrate or the heater is used under the condition of applied mechanical load, the
10 occurrence probability of accidents such as cracks in the substrate 1 can be reduced.

 Next, with reference to Figs. 4 to 10, a method of producing the aluminum nitride substrate shown in Fig. 1 is described. Figure 4 shows a flow chart explaining the method of producing the substrate shown in Fig. 1.
15 Figure 5 is a schematic perspective view illustrating the jig used in the sintering step shown in Fig. 4. Figure 6 is a schematic plane view illustrating a state of the molded body mounted in the groove portion of the jig before the sintering step. Figure 7 is a schematic perspective view illustrating a state of piling up of the jig mounted with the molded body shown in Fig. 6. Figure 8
20 is a schematic sectional view illustrating the jig pile formed by piling up a plurality of jigs shown in Fig. 7. Figure 9 is an enlarged schematic view illustrating one of the jigs in the jig pile shown in Fig. 8. Figure 10 is a schematic perspective view illustrating a state of the jig pile shown in Fig. 8

The above-mentioned preparing step (S100) and the mixing step (S200) together correspond to the step for preparing the raw material.

Next, a molding step (S300) is conducted (Fig. 4). The molding step (S300) is to form or to prepare a sheet-shaped molded body and produces the
5 sheet form molded body that is to be the substrate 1 (Fig. 1). Herein, conventional methods such as doctor-blade method, extrusion, roll-compaction method, etc. may be used to prepare the molded body.

Next, a drying step (S400) is conducted (Fig. 4), in which natural drying of a molded body is performed in a state where the molded body is
10 placed on a mesh tray made of stainless steel with a flat surface.

The time used for the drying step (S400) is not less than 1 hour. The natural drying time is preferably not less than 10 hours, and more preferably 20 hours or more. Also, such natural drying step is preferably conducted at the atmospheric temperature ranging from 0°C to 40°C, and more preferably
15 from 15°C to 25°C.

Under the above conditions, the solvent or moisture contained in the molded body can be volatilized out of the entire portion of the molded body at a relatively low speed. As a result, contraction of the molded body due to the drying step can uniformly occur over the entire molded body. Accordingly,
20 substantially no deflection is generated within the sheet-shaped molded body, and it is possible to reduce the possible occurrence of warpage or waviness of the molded body or the sintered substrate in post-processes such as a binder removing step (S500) (Fig. 4) and a sintering step (S600) (Fig. 4).

REPLACED BY
PAT 3,412,107

Next, the binder removing step (S500) (Fig. 4) is conducted. In the binder removing step (S500) for removing the binder, naturally dried molded bodies are heated for a predetermined time in a state where each molded body is placed in a depressed portion 6 (Fig. 5) of a jig 5 (Fig. 5) which is used in the
5 sintering step (S600) described hereinafter. As a result, the binder can be volatilized and removed from the molded body. The heating conditions may be such that the heating temperature is within the range of 400°C to 900°C and the heating time is within the range of 5 hours to 200 hours.

Next, the sintering step (S600) (Fig. 4) is conducted. The heating
10 furnace such as an all-carbon furnace or an all-metal furnace, or a combination thereof may be used as a heating furnace used in the sintering step (S600). The heating furnace preferably is an all-metal furnace. The term "all-metal furnace" as used herein refers to a heating furnace whose heater, heating chamber and the like (except for the jig shown in Fig. 5) are constituted by a
15 high-melting point metal material such as molybdenum (Mo) or tungsten (W). By employing the all-metal furnace, it is possible to prevent the atmosphere in the heating chamber from changing to an atmosphere excessively containing carbon during the sintering step. On the other hand, when the all-carbon furnace (the furnace using a carbon based material for the heater or the
20 heating chamber as a constructional material in the heating furnace) is used for sintering, the atmosphere in the heating chamber is altered into the atmosphere containing carbon during the sintering step. And, oxygen derived from the molded body to be sintered reacts with carbon in the carbon-

Also, the jig pile having the jigs 5a to 5k and 5m to 5p is placed inside a metal case 11 for a case made of metal as shown in Fig. 10. Here, the metal case 11 comprises a main body 9 and a cover 10. The jig pile is inserted inside the main metal case body 9. Also, the cover 10 is arranged to close an opening portion for placing the jig pile having the jigs 5a to 5k and 5m to 5p inside the main metal case body 9. As a material to constitute the metal case 11, molybdenum (Mo), for example, may be used.

And, in a state where the metal case is placed inside the heating furnace, sintering of the molded bodies 22 is performed (Fig. 8). As a result, it is possible to better reduce the risk of excessive inflow of the atmospheric gas around the molded bodies 22 (Fig. 8). Accordingly, it is possible to sinter the molded bodies 22 in a state where almost no warpage or the like is caused.

Also, although the thickness of the substrate 1 according to the present invention is set as 2 mm or less in the above description, by applying the production method as described above, almost no warpage is generated even if the thickness T of the substrate 1 (Fig. 1) is 1 mm or less. Typically, the thinner the thickness T the more prone to generate warpage in the substrate 1. However, the generation of such warpage can be prevented by the present invention.

Next, as shown in Fig. 4, a polishing step (S700) is conducted. In the polishing step (S700), by polishing the surface of the substrate comprising the aluminum nitride sintered body obtained in the sintering step (S600), a desired thickness of the surface is removed. In this way, the substrate 1 as

shown in Fig. 1 can be obtained.

Further, surface polishing allowance (chipping allowance) of the substrate obtained in the polishing step (S700) may be 10 μm or less at the surface of one side thereof. It is possible to have this value because warpage or waviness of the substrate 1 after the sintering step (S600) is small in the method of producing the substrate 1 according to the present invention. In other words, since the substrate 1 of the present invention shows sufficiently small warpage or waviness even immediately after the sintering step (S600), it is possible to sufficiently reduce the chipping allowance that is needed to obtain the substrate having the required flatness (value of warpage or waviness height). Furthermore, such polishing step may optionally be eliminated, depending on the applications of the substrate 1.

On the other hand, in the case of a substrate exhibiting relatively large warpage or waviness immediately after the sintering step (S600), the sintering step (S600) has been conducted with the substrate having a larger thickness and the chipping allowance has been increased so as to obtain the required flatness. In such case, since the chipping allowance is increased, it leads to increases of the time required for production and material cost, etc. In contrast, the method for producing the aluminum nitride substrate according to the present invention can reduce the production cost.

In the polishing step (S700), it is also possible to polish a surface of the sintered substrate by a peripheral circumference of a rotating body having a form of a deformable circular column (or cylindrical form) or having a disc

range of 0.01 wt.% to 1 wt.% in terms of the corresponding elements.

Group 2A elements or compounds thereof and Group 3A elements or compounds thereof typically function as a sintering aid to promote sintering of aluminum nitride which is a difficult material to be sintered. That is, the
5 elements or compounds thereof form a liquid phase by a reaction of the elements or compounds thereof with oxides (alumina) formed on the surface of particles of the aluminum nitride powder which is the major material of the substrate 1 (Fig. 1). The liquid phase binds the aluminum nitride particles together and consequently promotes the sintering of aluminum nitride. The
10 content of the above elements or compounds thereof may be within a range required for a conventional sintering aid. More specifically, the content of the above elements or compounds thereof is preferably in the range of 0.1 wt.% to 10 wt.% as a total in terms of the element.

In addition, in the substrate 1, the aluminum nitride constituting the
15 substrate 1 preferably has a small particle diameter. If the particle diameter is small, it is possible to accomplish a uniform and dense distribution of the aid component deposited on the surface of the sintered substrate 1. As a result, when the heating element and the electrode or the like are formed on the surface of the substrate 1, it is possible to obtain improved adhesiveness
20 between the surface of the substrate 1 and the heating element and electrode part, respectively.

On the other hand, if the aluminum nitride particle diameter is relatively large, the surface roughness of the substrate 1 increases. Thus, in

In such a case, the elements used in the sintering aid preferably comprise calcium (Ca) of Group 2A and yttrium (Y), neodymium (Nd) and ytterbium (Yb) of Group 3A, or compounds thereof. Particularly, it is preferable to use the elements described above in combination. By employing
5 the sintering aid comprising these elements, the sintering temperature in the sintering step (S600) (Fig. 4) can be decreased to 1,800°C or less. As a result, it is possible to prepare the aluminum nitride particles in the substrate 1 (Fig. 1) with an average particle diameter 4.0 μm or less.

(Second embodiment)

10 Figure 11 is a schematic perspective view illustrating a metallized substrate according to the present invention. With reference to Fig. 11, the metallized substrate of the present invention will be described.

As shown in Fig. 11, the metallized substrate 12 comprises an aluminum nitride substrate 1 (substrate 1) consisting of an aluminum nitride
15 sintered body made of aluminum nitride as a major component and a metallized layer 13 formed on the surface of the substrate 1. The substrate 1 may be the aluminum nitride substrate of the first embodiment according to the present invention shown in Fig. 1. The metallized substrate 12 has the warpage ranging from 0 $\mu\text{m}/\text{mm}$ to 5 $\mu\text{m}/\text{mm}$. The definition of the warpage
20 is identical to that in the substrate 1 of the first embodiment.

In this manner, by forming the metallized substrate 12 using the substrate 1 with a large size having the maximum length ML (Fig. 1) not less than 320 mm shown in Fig. 1, it is possible to manufacture electronic parts in

insulating properties may be used. This glass layer may contain each oxide of zinc, silicon, lead and manganese. Also, the contents of the elements contained in the glass layer are preferably in the range of 50 wt.% to 85 wt.% of zinc in terms of ZnO, 5.0 wt.% to 30 wt.% of silicon in terms of SiO₂, 3.0
5 wt.% to 15 wt.% of lead in terms of PbO, and 1.0 wt.% to 10 wt.% of manganese in terms of MnO.

All of the oxides of the respective elements such as zinc, silicon, lead and manganese show favorable wettability to the aluminum nitride sintered body containing each compound of calcium, ytterbium and neodymium.
10 Therefore, when any of the above oxides is applied to the insulating layer 17 (Fig. 13), it is possible to realize excellent adhesiveness between the insulating layer 17 and the substrate 1 consisting of the aluminum nitride sintered body.

Further, the insulating layer 17 comprising the above oxides has the thermal expansion coefficient of 3.7×10^{-6} to 5.0×10^{-6} , which is relatively close
15 to the thermal expansion coefficient of the aluminum nitride sintered body. As a result, the generation of warpage in the substrate 1 can be reduced when the insulating layer 17 is formed.

Also, in the case where the insulating layer 17 has a composition of components within the range as described above, if a material comprising
20 silver or a silver-lead alloy as the major component is employed for the heating element 16, it is possible to perform firing of the insulating layer 17 at a temperature lower by about 100°C than the firing temperature of the heating element 16 typically ranging from 800°C to 900°C, that is, around 700°C.

Examples

(Example 1)

In order to confirm the effect of the present invention, samples were prepared as follows and were subjected to various measurements.

- 5 First, the raw materials 1 to 3 containing the components listed in the following Table I were prepared.

Table I

Name of raw material	Major raw material AlN	(wt.%)					
		Aid Y ₂ O ₃	Aid Nd ₂ O ₃	Aid Yb ₂ O ₃	Aid Al ₂ O ₃	Aid SiO ₂	Aid CaO
Raw material -1	97.00	3.00	0.00	0.00	0.00	0.00	0.00
Raw material -2	96.80	0.00	0.90	1.00	0.90	0.20	0.20
Raw material -3	98.48	0.00	1.00	0.50	0.00	0.00	0.02

- 10 By using the above raw materials 1 to 3, a total of 69 samples from No. 1 to No. 69 shown in Tables II to IV were prepared by the production method described in the first embodiment of the present invention. The samples have different sizes of the substrates (length, width, thickness of the substrates), drying times in the drying step (S400) (Fig. 4), and volume ratios in a closed
- 15 space during the sintering step (S600) (Fig. 4) (that is, a ratio of the volume of the molded body 22 to a volume of the space 8 shown in Fig. 9) as listed in Tables II to IV. Other production conditions of each sample are described as follows also.

In the raw material preparation step (S100) (Fig. 4), the raw materials 1 to 3 shown in Table I were prepared. Then, the mixing step (S200) (Fig. 4) was conducted to blend the components of each of the raw materials 1 to 3 with solvents. In the molding step (S300) (Fig. 4), the three mixtures from the raw materials 1 to 3 were formed into sheet-shaped molded bodies that are to be the samples No. 1 to No. 69. The doctor-blade method was adopted for preparing thin molded bodies having a thickness of less than 1 mm, while an extrusion method was used for preparing thicker molded bodies having a thickness not less than 1 mm.

Next, in the drying step (S400) (Fig. 4), the molded bodies that are to be the samples were subjected to natural drying for the drying times shown in Tables II to IV. Next, the jigs made of boron nitride (BN) shown in Fig. 5 were prepared for the respective samples such that the depressed portions of the jigs were formed to satisfy the closed space volume ratios shown in Tables II to IV. Each of the molded bodies was placed in each of the jigs. And the binder removing step (S500) (Fig. 4) was conducted under a condition of the heating temperature of 850°C and nitrogen atmosphere. In addition, the heating temperature in the range of 400°C to 900°C can be used.

Next, in the sintering step (S600) (Fig. 4), each of the molded bodies after the binder removing step (S500) was subjected to sintering for 10 hours under the conditions of a heating temperature of 1,700°C, the atmospheric pressure controlled to normal pressure and nitrogen atmosphere. The sintering time may be in the range of 2 hours to 30 hours.

Table II

Sample No.	Name of raw material	Length (mm)	Width (mm)	Maximum length (ML) (mm)	Thickness (mm)	Drying time (hr)	Closed space volume ratio (%)	Warpage (μm/mm)	Waviness (μm)	Crack
1	Raw material - 1	409	95	420	0.6	20	50	0.17	26	No
2*		409	95	420	0.6	0.5	50	2.26	110	Yes
3		409	95	420	0.6	2	50	1.9	71	No
4		409	95	420	0.6	5	50	1.19	62	No
5		409	95	420	0.6	15	60	0.48	35	No
6		409	95	420	0.6	25	50	0.19	20	No
7*		409	95	420	0.6	20	5	2.19	96	Yes
8		409	95	420	0.6	20	15	1.9	74	No
9		409	95	420	0.6	20	25	1.79	53	No
10		409	95	420	0.6	20	65	0.24	31	No
11*		409	95	420	0.6	20	90	2.02	76	Yes
12*		409	95	420	1.8	0.5	5	2.05	95	Yes
13*		409	95	420	1.2	0.5	5	2.21	103	Yes
14*		409	95	420	0.6	0.5	5	2.98	115	Yes
15*		409	95	420	1.8	0.5	90	2.02	78	Yes
16*		409	95	420	1.2	0.5	90	2.33	83	Yes
17*		409	95	420	0.6	0.5	90	2.64	94	Yes
18		368	95	380	0.6	20	50	0.16	27	No
19		337	95	350	0.6	20	50	0.14	26	No
20		311	95	325	0.6	20	50	0.12	21	No
21*		368	95	380	0.6	20	5	2.21	78	Yes
22*		337	95	350	0.6	20	5	2.29	86	Yes
23*		311	95	325	0.6	20	5	2.06	94	Yes

* Comparative examples

Table III

Sample No.	Name of raw material	Length (mm)	Width (mm)	Maximum length (ML) (mm)	Thickness (mm)	Drying time (hr)	Closed space volume ratio (%)	Warpage (µm/mm)	Waviness (µm)	Crack
24	Raw material - 2	409	95	420	0.6	20	50	0.14	24	No
25*		409	95	420	0.6	0.5	50	2.24	115	Yes
26		409	95	420	0.6	2	50	1.86	73	No
27		409	95	420	0.6	5	50	1.07	58	No
28		409	95	420	0.6	15	50	0.48	32	No
29		409	95	420	0.6	25	50	0.21	21	No
30*		409	95	420	0.6	20	5	2.31	98	Yes
31		409	95	420	0.6	20	15	1.86	62	No
32		409	95	420	0.6	20	25	1.71	53	No
33		409	95	420	0.6	20	65	0.21	42	No
34*		409	95	420	0.6	20	90	2.14	77	Yes
35*		409	95	420	1.8	0.5	5	2	100	Yes
36*		409	95	420	1.2	0.5	5	2.24	118	Yes
37*		409	95	420	0.6	0.5	5	3.24	130	Yes
38*		409	95	420	1.8	0.5	90	2.19	88	Yes
39*		409	95	420	1.2	0.5	90	2.52	93	Yes
40*		409	95	420	0.6	0.5	90	2.74	101	Yes
41		368	95	380	0.6	20	50	0.13	25	No
42		337	95	350	0.6	20	50	0.11	24	No
43		311	95	325	0.6	20	50	0.12	19	No
44*		368	95	380	0.6	20	5	2.42	97	Yes
45*		337	95	350	0.6	20	5	2.4	83	Yes
46*		311	95	325	0.6	20	5	2.49	78	Yes

* Comparative examples

Table IV

Sample No.	Name of raw material	Length (mm)	Width (mm)	Maximum length (ML) (mm)	Thickness (mm)	Drying time (hr)	Closed space volume ratio (%)	Warpage (µm/mm)	Waviness (µm)	Crack
47	Raw material - 3	409	95	420	0.6	20	50	0.17	20	No
48*		409	95	420	0.6	0.5	50	2.33	123	Yes
49		409	95	420	0.6	2	50	1.98	72	No
50		409	95	420	0.6	5	50	1.29	63	No
51		409	95	420	0.6	15	50	0.67	27	No
52		409	95	420	0.6	25	50	0.17	26	No
53*		409	95	420	0.6	20	5	2.38	79	Yes
54		409	95	420	0.6	20	15	1.95	60	No
55		409	95	420	0.6	20	25	1.43	51	No
56		409	95	420	0.6	20	65	2.38	40	No
57*		409	95	420	0.6	20	90	2.19	76	Yes
58*		409	95	420	1.8	0.5	5	2.26	115	Yes
59*		409	95	420	1.2	0.5	5	2.69	122	Yes
60*		409	95	420	0.6	0.5	5	3.33	132	Yes
61*		409	95	420	1.8	0.5	90	2.21	100	Yes
62*		409	95	420	1.2	0.5	90	2.71	95	Yes
63*		409	95	420	0.6	0.5	90	2.9	86	Yes
64		368	95	380	0.6	20	50	0.21	18	No
65		337	95	350	0.6	20	50	0.2	17	No
66		311	95	325	0.6	20	50	0.18	15	No
67*		368	95	380	0.6	20	5	2.42	94	Yes
68*		337	95	350	0.6	20	5	2.23	85	Yes
69*		311	95	325	0.6	20	5	2.09	78	Yes

* Comparative examples

REPLACED BY
PART 34 APT

Herein, the samples with * shown in Tables II to IV are the samples of comparative examples. On the contrary, the samples without * are the samples of examples according to the present invention. As seen from Tables II to IV, it is clear that the samples of the present invention have sufficiently small warpage and waviness.

(Example 2)

With respect to the samples No. 1 to No. 69 shown in Tables II to IV, the electrode part and the heating element were prepared on the surface of each sample as shown in Figs. 12 and 13 by using the screen-printing method.

More specifically, by using the screen-printing method as shown Fig. 14, a silver-platinum (Ag-Pt) paste was applied to the portions where electrode parts 15a and 15b (Fig. 12) were to be formed, and a silver-palladium (Ag-Pd) paste was applied to the portion in which the heating element 16 was to be formed.

Figure 14 is a schematic sectional view to explain the screen-printing method used to form the heating element and the electrode part on the surface of each sample, that is, the substrate. As shown in Fig. 14, each sample (substrate 1) is placed on top of the upper surface of a base 18, then a screen 19 is placed on the substrate 1. On the screen 19, patterns for the heating element, the electrode part, etc. are formed beforehand. The paste to constitute the electrode parts 15a and 15b (Fig. 12) or the heating element 16 (Fig. 12) is placed on the screen 19, and a squeegee 20 is moved in the direction indicated by an arrow 21 while pressing the squeegee 20 toward the

substrate 1. As a result, the Ag-Pt paste or the Ag-Pd paste can be supplied on the surface of the substrate 1 with a predetermined pattern through the screen 19. And, by conducting a desired heat-treatment, the electrode parts 15a and 15b and the heating element 16 (Fig. 12) can be formed on the surface
5 of the substrate 1.

When the screen-printing method is conducted as described above, cracks may be generated in the sample if warpage or waviness is present on the sample (substrate 1). Therefore, the generation of cracks on the sample due to the screen-printing method was examined for all of the samples. The
10 results are shown in the "crack" column in Tables II to IV

As seen in Tables II to IV, cracks were found in the samples (the sintered bodies) having the warpage of 2 $\mu\text{m}/\text{mm}$ or more or the waviness of 76 μm or more. And, there was no generation of cracks in the samples of the examples of the present invention even after carrying out the screen-printing
15 method since all of these samples had sufficiently small warpage and waviness. (Example 3)

With respect to the samples Nos. 1, 24 and 47 among the samples listed in Tables II to IV, the thermal conductivity was measured for each sample before performing screen-printing of the pastes to be formed into the
20 electrode parts 15a and 15b (Fig. 12) and the heating element 16 (Fig. 12) on the surface of the sample. The measurement was performed by the laser-flashing method. Then, the Ag-Pt paste and Ag-Pd paste were introduced on the surfaces of the above-mentioned samples Nos. 1, 24 and 47 according to a

shown in Table V.

Table V

Name of raw material	Sample No.	Thermal conductivity (W/m ·K)	Softening/melting of solder
Raw material – 1	1	180	Partial melting
Raw material – 2	24	95	None
Raw material – 3	47	130	Softening

As seen also in Table V, since the values of thermal conductivity of the
5 samples Nos. 1 and 47 using the raw material-1 and the raw material-3,
respectively, were relatively high, a softened portion and a partially molten
portion were observed in these samples at the connection between the
electrode parts and the lead wires. On the contrary, since the thermal
conductivity of the sample No. 24 using the raw material-2 was relatively low,
10 softening or melting did not occur at the connection between the electrode
parts and the lead wires.

The above preferred embodiments and examples disclosed in this
document are only illustrative and not intended to limit the scope of the
invention. The scope of the present invention is not defined by the
15 embodiments or examples described above, but it is defined by the claims, and
it is intended to include all the modifications within the meaning and range
that are equivalent to the scope of the claims.

Industrial applicability

20 As described above, an aluminum nitride sintered body having a large
area and a small thickness compared to conventional ones, and also having

CLAIMS

1. An aluminum nitride sintered body having a maximum length of 320 mm or more, a thickness of more than 0 mm and 2 mm or less, a warpage of 0 $\mu\text{m}/\text{mm}$ or more and less than 2 $\mu\text{m}/\text{mm}$, and a local waviness height of 0 μm or more and 100 μm or less.

2. The aluminum nitride sintered body according to claim 1, wherein the aluminum nitride sintered body has the thermal conductivity of 50 W/m·K or more and 250 W/m·K or less.

3. A metallized substrate comprising a plate-shaped substrate made of the aluminum nitride sintered body according to claim 1 or 2 and an electrically conductive metallized layer formed on at least a part of the surface of the substrate.

4. A metallized substrate comprising a substrate including an aluminum nitride sintered body having a maximum length of 320 mm or more, a thickness of more than 0 mm and 2 mm or less, and a local waviness height of 0 μm or more and 100 μm or less, and an electrically conductive metallized layer formed on at least a part of the surface of the substrate, wherein a warpage of the metallized substrate is in the range of 0 $\mu\text{m}/\text{mm}$ to 5 $\mu\text{m}/\text{mm}$.

5. A heater comprising the metallized substrate according to claim 3 or 4, an electrode part arranged on the surface of the substrate and connected to the metallized layer, and an insulating layer arranged on the metallized layer.

6. A method of producing an aluminum nitride sintered body, comprising the steps of:

preparing a raw material containing a binder and a major material of aluminum nitride;

5 forming a sheet-shaped molded body using the raw material;

drying the molded body for 1 hour or more;

removing the binder from the molded body that has been subjected to the drying step; and

sintering the molded body free of the binder.

10 7. A method of producing the aluminum sintered body according to claim 6, wherein the sintering step is performed by sintering the molded body arranged in a space surrounded by a jig comprising boron nitride as a major component, and wherein the ratio of the volume of the molded body before sintering to the volume of the space in the range from 10% to 70%.

15 8. A method of producing an aluminum nitride sintered body, comprising the steps of:

preparing a sheet-shaped molded body containing aluminum nitride as a major component; and

20 sintering the molded body by arranging a piece of the molded body in the space surrounded by a jig comprising boron nitride as a major component.

9. A method of producing an aluminum nitride sintered body according to claim 8, wherein the ratio of the volume of the molded body before sintering to the volume of the space in the range from 10% to 70%.

10. A method of producing an aluminum nitride sintered body according to any one of claims 7 to 9, wherein the jig has a depressed portion on the top surface of a flat plate-shaped base to arrange the molded body therein, and in the sintering step, sintering is performed in a state where a
5 plurality of the jigs each having the molded body in the depressed portion thereof are piled up.

11. A method of producing an aluminum nitride sintered body according to claim 10, wherein the sintering step is performed in a state where a jig pile formed by stacking the plurality of jigs is placed inside of a case made
10 of a metal material.

12. A jig used in a sintering step to produce an aluminum nitride sintered body, wherein the jig comprises a flat plate-shaped base containing boron nitride and having a depressed portion formed on the surface thereof to arrange a molded body that is to be the aluminum nitride sintered body,
15 wherein dimensions of the depressed portion are determined such that the ratio of the volume of the molded body to the volume of the depressed portion is within the range of 10% to 70%.